

What is claimed:

1. A method for separating a tocopherol from a first tocopherol admixture comprising at least one tocopherol, a first fatty acid, and an esterifying compound, comprising
  - (a) heating the first tocopherol admixture comprising the tocopherol, the first fatty acid, and the esterifying compound for a sufficient time and temperature to substantially esterify the first fatty acid with the esterifying compound to produce a second tocopherol admixture comprising the tocopherol, esterified first fatty acid, and unesterified first fatty acid;
  - (b) distilling the second tocopherol admixture for a sufficient time and temperature to substantially remove the unesterified first fatty acid from the second tocopherol admixture to produce a third tocopherol admixture comprising the tocopherol and the esterified first fatty acid, with substantially removed unesterified first fatty acid;
  - (c) distilling the third tocopherol admixture for a sufficient time and temperature to substantially remove the tocopherol from the third tocopherol admixture to produce a fourth tocopherol admixture comprising the removed tocopherol and a non-tocol component; and
  - (d) extracting the tocopherol from the fourth tocopherol admixture with an extraction solvent comprising a polar, organic solvent that is miscible with water to produce a two phase system comprising a first phase containing a majority of the extraction solvent and a second phase, wherein the selectivity of the extraction solvent for the tocopherol with

respect to the non-tocol component is greater than unity, and removing the first phase from the second phase,

with the proviso that the extraction solvent is not a neat alcohol,

wherein step (b) can be conducted before step (c), or step (c) can be conducted before step (b), wherein steps (b) and (c) are conducted after step (a) and prior to step (d).

2. The method of Claim 1, wherein the extraction solvent does not comprise an alcohol.
3. The method of Claim 1, wherein the tocopherol is gamma-tocopherol.
4. The method of Claim 1, wherein the esterifying compound comprises a sterol ester of a fatty acid, sterol, triterpenoid alcohol, methyl-sterol, monoglyceride, diglyceride or triglyceride or a combination thereof.
5. The method of Claim 1, wherein the heating step (a) is conducted at a temperature of from 70 to 300°C and at a pressure of from 20 to 760 torr.
6. The method of Claim 1, wherein the heating step (a) is conducted at a temperature of from 150 to 230°C and at a pressure of from 20 to 200 torr.
7. The method of Claim 1, further comprising removing water during the heating step.
8. The method of Claim 1, further comprising adding a catalyst to the first tocopherol admixture prior to the heating step.

9. The method of Claim 8, wherein the catalyst comprises an alkyl tin compound, a zinc salt of an organic acid, zinc oxide, a titanium (IV) alkoxide, or a mineral acid, or a combination thereof.
10. The method of Claim 1, further comprising adding a second fatty acid to the first tocopherol admixture prior to heating step (a).
11. The method of Claim 10, wherein the second fatty acid comprises a  $C_{14}$  to  $C_{22}$  fatty acid.
12. The method of Claim 1, wherein prior to the heating step (a), an alcohol is not added to the first tocopherol admixture.
13. The method of Claim 1, wherein the second tocopherol admixture is distilled in step (b) at a temperature of from 125 to 320°C at a pressure of from 0.01 to 10 torr.
14. The method of Claim 1, wherein the second tocopherol admixture is distilled in step (b) at a temperature of from 150 to 200°C at a pressure of from 0.01 to 10 torr.
15. The method of Claim 1, wherein after the distillation step (b) and prior to the distillation step (c), further distilling the third tocopherol admixture at a temperature of from 150 to 300°C at a pressure of from 0.01 to 5 torr.
16. The method of Claim 1, wherein the third tocopherol admixture is distilled in step (c) at a temperature of from 170 to 270°C at a pressure of from 0.005 to 2 torr.
17. The method of Claim 1, wherein steps (a)-(c) are conducted in series.

18. The method of Claim 1, wherein steps (a)-(c) are conducted at from 1 to 24 hours.
19. The method of Claim 1, wherein steps (a)-(c) are conducted at from 2 to 10 hours.
20. The method of Claim 1, wherein the fourth tocopherol admixture has from 1 to 50 % by weight tocopherols.
21. The method of Claim 1, wherein the extraction solvent is an aqueous composition.
22. The method of Claim 1, wherein the extraction solvent comprises acetic acid, aqueous acetic acid, propionic acid, aqueous propionic acid, acetone, aqueous acetone, aqueous methanol, aqueous ethanol, acetonitrile, aqueous acetonitrile, aqueous 2-methoxyethanol, aqueous 2-ethoxyethanol, aqueous 2-propoxyethanol, aqueous isopropanol, 1,4-dioxane, dimethylacetamide, dimethylformamide, N-methyl pyrrolidinone, butadiene sulfone, dimethyl sulfoxide, 2-methoxyethyl ether, dimethoxyethane, or the aqueous solvent thereof, or a combination thereof.
23. The method of Claim 1, wherein the extraction solvent comprises acetonitrile, aqueous acetonitrile, aqueous ethanol, acetic acid, aqueous acetic acid, aqueous isopropanol, aqueous propionic acid, or a combination thereof.
24. The method of Claim 1, wherein the extraction solvent comprises aqueous acetonitrile.
25. The method of Claim 1, wherein the extraction solvent comprises aqueous acetonitrile, and the aqueous acetonitrile comprises from 0.5 to 20 % by weight

water and from 80 to 99.5 % by weight acetonitrile, wherein the sum of the acetonitrile and water is equal to 100%.

26. The method of Claim 1, wherein the extraction solvent comprises aqueous ethanol.
27. The method of Claim 1, wherein the extraction solvent comprises aqueous ethanol, and the aqueous ethanol comprises from 0.5 to 20 % by weight water and from 80 to 99.5 % by weight ethanol, wherein the sum of the ethanol and water is equal to 100%.
28. The method of Claim 1, wherein the extraction solvent comprises aqueous acetic acid.
29. The method of Claim 1, wherein the extraction solvent comprises aqueous acetic acid, and the aqueous acetic acid comprises from 1 to 15 % by weight water and from 85 to 99 % by weight acetic acid, wherein the sum of the acetic acid and water is equal to 100%.
30. The method of Claim 1, wherein the extraction step is conducted at from 20 to 55°C.
31. The method of Claim 1, wherein the extraction step is a continuous, countercurrent extraction.
32. The method of Claim 1, wherein the extraction solvent to feed ratio is from 20:1 to 1:20.

33. The method of Claim 1, further comprising after removing the first phase from the second phase, removing the extraction solvent from the first phase by evaporation or distillation.
34. The method of Claim 1, wherein the first tocopherol admixture comprises vegetable oil deodorizer distillate.
35. The method of Claim 34, wherein the vegetable oil deodorizer distillate comprises canola oil, sunflower oil, or soybean oil deodorizer distillate.
36. The method of Claim 1, wherein the first tocopherol admixture is soybean oil deodorizer distillate.
37. The method of Claim 1, wherein the first tocopherol admixture is sunflower oil deodorizer distillate.
38. The method of Claim 1, wherein (1) the first tocopherol admixture comprises soybean oil deodorizer distillate or sunflower oil deodorizer distillate; (2) the extraction solvent comprises aqueous acetonitrile comprising 0.5 to 20% by weight water and from 80 to 99.5% by weight acetonitrile; and (3) the extraction step is continuous and countercurrent.
39. A method for separating tocopherol from a tocopherol admixture comprising at least one tocopherol and at least one non-tocol component, wherein the amount of the tocopherol in the tocopherol admixture is from 10 to 55% by weight of the tocopherol mixture and the amount of fatty acid in the tocopherol admixture is less than 5%, comprising extracting tocopherol from the tocopherol admixture with an extraction solvent comprising a polar, organic solvent that is miscible with water to produce a two phase system comprising a first phase containing a majority of the extraction solvent and a second phase, wherein the

selectivity of the extraction solvent for the tocopherol with respect to the non-tocol component is greater than unity, and removing the first phase from the second phase, with the proviso that the extraction solvent does not comprise a neat alcohol.

40. A composition produced by the process of Claim 1.
41. A composition produced by the process of Claim 25.
42. A composition produced by the process of Claim 38.
43. A method for separating a tocopherol from a first tocopherol admixture comprising at least one tocopherol, a first fatty acid, and an esterifying compound, comprising
  - (a) heating the first tocopherol admixture comprising the tocopherol, the first fatty acid, and the esterifying compound for a sufficient time and temperature to substantially esterify the first fatty acid with the esterifying compound to produce a second tocopherol admixture comprising the tocopherol, esterified first fatty acid, and unesterified first fatty acid;
  - (b) distilling the second tocopherol admixture for a sufficient time and temperature to substantially remove the unesterified first fatty acid from the second tocopherol admixture to produce a third tocopherol admixture comprising the tocopherol and the esterified first fatty acid, with substantially removed unesterified first fatty acid;
  - (c) distilling the third tocopherol admixture for a sufficient time and temperature to substantially remove the tocopherol from the third

tocopherol admixture to produce a fourth tocopherol admixture comprising the removed tocopherol and a non-tocol component; and

- (d) extracting the tocopherol from the fourth tocopherol admixture with an extraction solvent comprising a polar, organic solvent that is miscible with water to produce a two phase system comprising a first phase containing a majority of the extraction solvent and a second phase, wherein the selectivity of the extraction solvent for the tocopherol with respect to the non-tocol component is greater than unity, and removing the first phase from the second phase,

with the proviso that the extraction solvent is not a neat alcohol,

wherein step (b) can be conducted before step (c), or step (c) can be conducted before step (b), wherein steps (b) and (c) are conducted after step (a) and prior to step (d),

wherein the first tocopherol admixture is soybean oil, and the extraction solvent comprises acetic acid, aqueous acetic acid, propionic acid, aqueous propionic acid, acetone, aqueous acetone, 1,4-dioxane, aqueous 1,4-dioxane, dimethylacetamide, aqueous dimethylacetamide, dimethylformamide, aqueous dimethylformamide, N-methyl pyrrolidinone, aqueous N-methyl pyrrolidinone, butadiene sulfone, aqueous butadiene sulfone, dimethyl sulfoxide, aqueous dimethyl sulfoxide, 2-methoxyethyl ether, aqueous 2-methoxyethyl ether, dimethoxyethane, aqueous dimethoxyethane, aqueous methanol, aqueous ethanol, aqueous acetonitrile, or a combination thereof.

44. The method of Claim 43, wherein the extraction solvent is aqueous acetonitrile.



45. A method for separating a tocopherol from a first tocopherol admixture comprising at least one tocopherol, a first fatty acid, and an esterifying compound, comprising
- (a) heating the first tocopherol admixture comprising the tocopherol, the first fatty acid, and the esterifying compound for a sufficient time and temperature to substantially esterify the first fatty acid with the esterifying compound to produce a second tocopherol admixture comprising the tocopherol, esterified first fatty acid, and unesterified first fatty acid;
  - (b) distilling the second tocopherol admixture for a sufficient time and temperature to substantially remove the unesterified first fatty acid from the second tocopherol admixture to produce a third tocopherol admixture comprising the tocopherol and the esterified first fatty acid, with substantially removed unesterified first fatty acid;
  - (c) distilling the third tocopherol admixture for a sufficient time and temperature to substantially remove the tocopherol from the third tocopherol admixture to produce a fourth tocopherol admixture comprising the removed tocopherol and a non-tocol component; and
  - (d) extracting the tocopherol from the fourth tocopherol admixture with an extraction solvent comprising a polar, organic solvent that is miscible with water to produce a two phase system comprising a first phase containing a majority of the extraction solvent and a second phase, wherein the selectivity of the extraction solvent for the tocopherol with respect to the non-tocol component is greater than unity, and removing the first phase from the second phase,

**ATTORNEY DOCKET NO.: 05015.0289**

with the proviso that the extraction solvent is not a neat alcohol,

wherein step (b) can be conducted before step (c), or step (c) can be conducted before step (b), wherein steps (b) and (c) are conducted after step (a) and prior to step (d),

wherein the first tocopherol admixture comprises soybean oil, sunflower oil, or canola oil, and the extraction solvent comprises acetone, aqueous acetone, or aqueous ethanol.

46. The method of Claim 45, wherein the extraction solvent comprises, acetone, aqueous acetone, aqueous ethanol, or a combination thereof.
47. The method of Claim 45, wherein the extraction solvent is aqueous ethanol.